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Characterisation of microstructure and creep properties of alloy 617 for high-temperature applications

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ABSTRACT

Current energy drivers are pushing research in power generation materials towards improved efficiency and improved environmental impact. In the context of new generation ultra-supercritical (USC) power plant, this is represented by increased efficiency, service temperature reaching 750 °C, pressures in the range of 35–37.5 MPa and associated carbon capture technology. Ni base alloys are primary candidate materials for long term high temperature applications such as boilers. The transition from their current applications, which have required lower exposure times and milder corrosive environments, requires the investigation of their microstructural evolution as a function of thermo-mechanical treatment and simulated service conditions, coupled with modelling activities that are able to forecast such microstructural changes. The lack of widespread microstructural data in this context for most nickel base alloys makes this type of investigation necessary and novel. Alloy INCONEL 617 is one of the Ni-base candidate materials. The microstructures of four specimens of this material crept at temperatures in the 650–750 °C range for up to 20,000 h have been characterised and quantified. Grain structure, precipitate type and location, precipitate volume fraction, size and inter-particle spacing have been determined. The data obtained are used both as input for and validation of a microstructurally-based CDM model for forecasting creep properties.

1. Introduction

The material requirements for USC applications, operating with steam target temperatures as high as 750 °C and pressures of 35–7.5 MPa, have brought forward the need for adapting and further developing existing materials, such as Ni-base superalloys. These have been used to date in relatively short term applications such as turbine engines in the aerospace industry. Forecasted requirements for USC applications have set the targets at minimum creep strength of 100 MPa at 100,000 h of service [1–7]. Since the microstructure of most of the candidate nickel base alloys has not been investigated after such long exposure periods of time, modelling activity has proven to be helpful in predicting the material microstructural response under simulated service conditions [8]. The superior combination of high temperature strength, corrosion/oxidation resistance and creep resistance make alloy INCONEL 617 (referred to as alloy 617) one of the Ni-base candidate materials. Alloy 617 is defined as a solid-solution strengthened material which is generally put in service in the solution annealed condition. However, the presence of γ′ and carbides, such as $M_{23}C6$, M_6C and MX in the as-received and in the aged conditions, plays a significant role in hardening the material [9–12] The present investigation concentrates on linking microstructural evolution of alloy 617 as a function of different thermo-mechanical treatments to the modelling activity, which forecasts creep behaviour, and comparing the predictions with experimental data. The microstructural analysis provides characterisation of the microstructural constituents and the quantification of phase fractions, mean particle size and inter-particle spacing (IPS). The quantitative data obtained serve as input and validation for the model predictions. The model combines a continuum damage mechanics (CDM) approach which has been successfully used for the prediction of creep rupture properties of 9 wt% – Cr ferritic steel [13], and which is now being adapted to the case of Ni-base superalloys systems.

2. Experimental procedure

2.1. Materials

The nominal chemical composition of the alloy studied in this investigation is listed in Table 1.

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Table 1
Composition of IN617.

Alloy	Chemical composition weight %												Ni
	C	Mn	Si	Al	Cr	Cu	Co	Mo	Fe	Ti	Nb+Ta	B	
IN617	0.05	–	–	0.08	20.0	–	10.0	8.0	–	–	–	–	Bal
	0.15	1.0	1.0	1.5	24.0	0.05	15.0	10.0	3.0	0.6	—	0.06	Bal

The 617 samples were investigated in the solubilised as-received (AR) and in the crept condition. The creep tests were not conducted to rupture. For phase quantification and modelling purposes, a fifth 617 specimen aged over a short period of time was also investigated. Table 2 summarises the thermo-mechanical treatment that the various specimens underwent before investigation.

2.2. Investigation procedure

Cutting of the samples was carried out using a Struers Accutom-5 cutting machine. Bakelite mounting for all samples was carried out in a Struers ProntoPress-10 machine. A Struers TegraPol-25 automatic polishing machine was used for both grinding and polishing. The latter was carried out with a 6 μm and finally 1 μm particle size diamond solution. Colloidal silica polishing was carried out for electron back-scatter diffraction (EBSD) analysis. Acetic glyceregia (a mixture of acetic acid, glycerine, HCl and HNO_3) was used as etching reagent for preparing 617 samples to reveal the microstructural features. A LEO 1530VP field emission gun scanning electron microscope (FEG-SEM) was used to image the sample surfaces. The most useful detection mode for this analysis was high resolution secondary electron referred to as In-lens. Energy dispersive X-ray analysis (EDX) was performed in the FEG-SEM system using an Oxford Instruments X-Max 80 mm^2 detector. Elemental mapping and point spectra were obtained for all the samples. EBSD analysis provided grain size and morphology. The system uses HKLNordlys F high speed Camera and Oxford Instrument Aztec EDX/EBSD microanalysis software for data collection. The grain size was also confirmed by using the mean linear intercept method. The hardness of all samples was measured by means of an Innovatest Nexus Series machine. A 10 kg load and 15 s dwell time were applied. For each sample, ten measurements were performed and the average hardness value determined. Finally, thermodynamic and kinetic calculations have been carried out with the thermo-kinetic software MatCalc (version 5.52) including the nickel thermodynamic database (version 2.00 prebeta 016) and diffusion database (version 2.00 prebeta 001).

2.3. Phase quantification, particle size and inter-particle spacing (IPS) analyses

In order to obtain statistically meaningful results, the phase quantification was performed on large areas imaged by means of FEG-SEM (In-lens). In order to validate the use of this technique, comparison was made with images obtained by means of TEM performed on samples obtained from another Ni-base alloy, nominally IN740 (Ni–0.03C–0.3Mn–0.5Si–0.9Al–25Cr–20Co–0.5Mo–0.7Fe–1.8Ti–2Nb+Ta) [14] as part of a similar phase quantification exercise. Montages of individual high magnification images were produced so that large areas could be analysed while retaining the ability of quantifying fine phases. Carbon replica were extracted for the large particles, such as MX and $M_{23}C_6$, whilst thin foils were produced specifically for the analysis of the γ′ particles. The morphology and dimension of the particles were comparable and therefore the FEGSEM imaging has been adopted as the main technique for phase imaging and quantification, the main advantages being the relative ease of sample preparation and the possibility of analysing very large areas, impossible to accomplish by using TEM. Fig. 1 shows MX and $M_{23}C_6$ particles imaged using TEM (a) and FEGSEM (b), precipitated at a grain boundary, having comparable dimensions.

Table 2
Sample specifications.

Specimen	Temperature (°C)	Load (MPa)	Time (h)
AR	–	–	–
1	650	130	19,299
2	700	110	20,764
3	700	94	20,168
4	750	68	16,075
5	750	–	100

Fig. 2 shows the reproducibility of γ′ particle size and dimensions, when they are imaged by means of TEM (a) and FEGSEM (b). Beside the many advantages of performing the quantification of γ′ by means of FEGSEM, there are associated inherent inaccuracies in the measurements.

The main source of error derives from the orientation of the precipitates which, when imaged in two dimensions, provide different results depending on what section of the precipitates is observed and measured. In this study the method consistently followed is based on three specific conditions:

- A large number of particles belonging to different sample areas are measured.
- The particles considered fall within the same threshold of contrast allowed by the In-lens detection mode, which suggests which regions of the particles close to the same plane of observation.
- In the case of cuboids, the areas of interest are chosen so that the sides of the particles are as parallel as possible to the observed plane.

The results obtained following this method have been compared to results obtained by means of TEM examination and with thermodynamic and kinetics calculations predicting the expected γ′ volume fractions as a function of isothermal exposure. The results obtained have proven to fit well with both TEM measurements and modelling predictions.

The tables in this article present results obtained by separately quantifying: all particles excluding γ′; γ′ particles; intergranular (GB) particles. The main reason for carrying out distinct analysis on different types of particles is mainly due to the average size of the particles. GB particles and γ′ particles are usually finer than the larger MX particles and require higher magnification imaging. Finally, grain boundaries considered for the analysis have been assumed to be representative for the majority of the microstructure. Fig. 3a shows a schematic of image processing carried out for the assessment of area fraction, particle size and IPS in the case of GB particles. The IPS is calculated following two different procedures and comparing the results. One is performed by considering the distance between two neighbouring particles along the grain boundary (see Fig. 3b). Another one is performed by